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The Electrochemistry and Properties of Poly-Paraphenylene Formed from the
Anodic Oxidation of Biphenyl in Aprotic Solvents

By

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THE ELECTROCHEMISTRY AND PROPERTIES OF
POLY-PARAPHENYLENE FORMED FROM
THE ANODIC OXIDATION OF BIPHENYL
IN APROTIC SOLVENTS

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ABSTRACT

Conducting poly-paraphenylene films can be grown on platinum electrodes via the electrochemical anodic polymerization of biphenyl. Electroreflectance and complex plane impedance analysis are used to investigate the conductivity of the polymer which, like polypyrrole, is high for the oxidized polymer and low for the neutral polymer. Films formed in this way are shown to have potential applications in batteries and are able to incorporate certain reactive species in a matrix that is useful for electrocatalysis.

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INTRODUCTION

In recent years there has been an enormous research effort in the field of conducting polymers and several have emerged with favorable properties. Notable examples[1] are polyphenylene sulphide (PPS), poly-paraphenylene (PPP), polypyrrole (PP), polyacetylene (PA) and recently polyaniline (PAN) and polyquinoline (PQ). Unfortunately, none of these can be easily recast from solution. This is a desirable feature in the design and fabrication of energy storage devices. It is also particularly desirable to be able to grow a conducting polymer electrochemically since electrochemistry provides a unique method for controlling film thickness, doping level, and so on. In this area much work has been carried out by Diaz and co-workers on PP[1b]. PPP on the other hand has received much less attention from the electrochemist. Indeed, with the exception of some interesting work by Rubinstein recently,[2,3] the authors know of no serious attempts to electrochemically grow the polymer. This reticence is undoubtedly because of the fact that the electrochemical oxidative polymerization of benzene requires the use of unattractive solvents such as HF or SO_2. Nevertheless the electrochemical growth of the polymer from more common solvents would certainly be desirable, since the homogeneously synthesized polymer has been shown to be readily doped to "p" or "n" type, with conductivities ranging over 17 orders of magnitude! The advantages of the fine control afforded by electrochemical methods for possible electronic applications therefore needs no discussion.

It is well known[4,5] that oligomers of benzene oxidize at increasingly lower potentials than the monomer. However the stability of the radical cation ($\varphi_n \cdot$) increases with chain length n, so longer chain precursors are less likely to polymerize. With these facts in mind we have chosen to investigate biphenyl (φ)

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as a possible precursor to polyphenylene. It should be pointed out that polyphenylene has a number of advantages over polypyrrole. for example, air stability.[6] It should also be noted that the cost of the parent compound (ϕ_2) is only 10% of that of pyrrole - a point for serious thought when considering possible commercial applications.

One problem which has been clearly apparent in the studies of electrochemically formed conducting polymers is the attenuated lifetimes of the polymers formed by these methods under potential cycling conditions. Poly-paraphenylene and Nafion films, however, have been shown to have considerably longer lifetimes when electrooxidized. We demonstrate in this work the ease of formation of dimensionally stable films of poly-paraphenylene by the electrochemical anodic oxidation of ϕ_2 dissolved in aprotic solvents. We use several complementary techniques to characterize the polymer, and discuss possible uses of the material thus formed as a support matrix for electrocatalysis and battery applications.

EXPERIMENTAL

In all experiments, potential control was achieved using a Hi-Tek Instruments DT 2101 potentiostat and PPR1 waveform generator. Frequency dispersion measurements were carried out using a Solartron 1250 frequency response analyzer and reflectance spectra were obtained in a conventional modulated specular reflectance (MSRS) configuration[7] using a Bentham 223 phase sensitive detector, 201E current amplifier and a RCA CA31000 photomultiplier. Infrared spectra were recorded using an IBM/Bruker IR98 FTIR spectrometer. Reagent grade chemicals were used and the acetonitrile (Burdick and Jackson

"Distilled in Glass" HPLC grade) was stored over alumina (Woelm Super Grade VI) prior to use. Potentials are referred to a Ag/Ag$^+$ (0.01M AgNO$_3$ in CH$_3$CN) reference electrode.

RESULTS

Polymer Film Properties

Figure 1 shows a cyclic voltammogram recorded at a platinum electrode on continued cycling in a 1.0 mM solution of ϕ_2. The results clearly indicate that an insulating surface film is formed, since the current on the second and successive cycles is seen to fall to only a fraction of the value observed on the first scan. The current cannot be recovered unless the electrode is mechanically polished. In a 1.0 M ϕ_2 solution however, the current was ohmically controlled at high potentials (Figure 2a), as evidenced by the linear tendency in the current-voltage response, and was accompanied by the appearance of two new redox couples centered at around 0.7 and 1.0V vs. Ag/Ag$^+$ on continued cycling. When the positive potential scan limit was reduced (Figure 2b), the magnitude of wave I was decreased in magnitude, but wave II remained constant and was not affected by stirring the solution. The loss of wave I indicates that this is a solution soluble species generated during the oxidation of ϕ_2. During this treatment, the electrode became covered with an adherent black film.

The voltammetry of the aforementioned film in a fresh solution containing only acetonitrile and supporting electrolyte was strongly dependent on the film growth conditions. Growth from dilute solutions tended to yield strongly adherent gold-colored films with characteristic voltammetry as shown in Figure 3a. Growth from concentrated solutions yielded loosely adherent black deposits

with voltammetry as depicted in Figure 3b. Note the persistence of wave II.
With both thick and thin films, an irreversible voltammetric peak was observed at
about +1.7V.

Under potentiostatic growth conditions the current transient indicated a
nucleation phenomenon (Figure 4) as evidenced by "rising" chronoamperometric
transients: at intermediate potentials, the current increases gradually as the
nucleation occurs. at which time there is a rapid catalytic increase in the
current due to the bulk deposition of PPP. It would thus appear that the
oxidation of ϕ_2 occurs preferentially on the polymer though we have no SEM data
to support this view as yet. Potentiostatically grown films displayed the same
sort of voltammetry as potentiodynamically grown films, the exact behavior
depending on the potential at which the polymer is formed and the ϕ_2
concentration. A common feature to all the films. however. was the existence of
an irreversible oxidation wave center at 1.7 V vs. Ag/Ag⁻, and involving ~ 10% of
the original "growth" charge.

The impedance of the polymer film as a function of time was measured by
conventional AC methods. Unlike Bard's studies on PP films[8a], we found that as
the layer thickened (under potentiostatic growth conditions) the film capacitance
steadily fell (Figure 5). In the case of polypyrrole. Bard has described a
capacitance which increases with film thickness. and has attributed this
phenomenon to the presence of pores.

Complex plane impedance analysis, an AC electrochemical technique that
provides the resistive and capacitative components of the cell impedance
directly, was performed on the PPP film (Figure 6). The results indicate that
when the electrode is held at increasingly positive potentials. both components
of the impedance are significantly reduced. and at potentials greater than 1.0 V

the impedance is purely capacitive. This phenomenon has been observed for the PP system previously[8b]. After "irreversible" oxidation at 2.0V, however, the impedance increased to the same value observed at the 0V level, and remained there even under an applied potential of 1.0V.

Further evidence for film conductivity comes from infrared reflectance spectroscopy. The IR reflectance spectrum for a neutral film is shown in Figure 7. The relative intensities of the $806cm^{-1}$ and $765cm^{-1}$ bands (unsubstituted and p-substituted phenyl C-H out-of-plane stretching band respectively) can be used to calculate the chain length.[9]. Also indicative of the chain length is the position of the C-H out-of-plane stretching band which varies from $837cm^{-1}$ for terphenyl to $806cm^{-1}$ for PPP.[10] From our data we calculate an average chain length of about 10 monomer units. When the film is oxidized most of the features are masked by the free carrier absorption, which again indicates a high film conductivity.

The polymer film was also characterized using modulated specular reflectance spectroscopy (MSRS) and spectra are shown in Figure 8. There are two absorption maxima centered at 620 and 380nm which increase in intensity as the average bias potential is made more positive to a maximum at around +500mV vs. Ag/Ag⁻. It was also possible to measure the UV absorption spectra of the polymer directly since it was found that the eléctrochemically grown polymer, unlike the homogeneously oxidized material.[3] is soluble in N-methyl pyrrolidone. (No other common laboratory solvents were found capable of dissolving the polymer.) The absorption spectrum. shown in Figure 9, exhibits peaks at 315nm. 330nm and 360nm. There is clearly a spectrum of oligomers with different chain lengths present. The 315nm peak probably corresponds to hexaphenyl[10] and the other maxima to correspondingly longer chains. Interestingly. the resultant solution

of oligomers could not be re-polymerized electrochemically. Similar behavior has been reported for oligomers of pyrrole.[11] This phenomenon is presumably because the radical cations formed in the oxidation of longer oligomers are more stable than θ_2^+.

Applications of the Film

An area of electrochemistry which shows great promise at present is electrocatalysis. Conductive polymers have a potential application here in that they can be used to trap a chemically reactive species and the reactivity of that species can be controlled electrochemically. Such an effect was discussed by ourselves[12] for a porphyrin trapped in PPP and later by Bard et al.[13] using polypyrrole as the support. There are a number of ways to trap the active center of the polymer. We have attempted to do this by simply generating the polymer in the presence of the species to be trapped. This approach appears to have worked quite well for a large number of species. Figure 10 shows a cyclic voltammogram. obtained in a solution containing only base electrolyte, of a PPP film electrodeposited in the presence of ferrocene. The ferrocene/ferrocenium couple is clearly observed (peaks A and B respectively), and persisted for several hundred scans without significant change. Interestingly, reduction wave VI (which is probably H⁻ reduction) was consistently larger for films containing ferrocene, since the neutral film is apparently less conducting. This probably indicates charge transfer mediation by the ferrocene.

Similar results were obtained for 9,10-diphenylanthracene and anthracene showing that quite bulky molecules can be trapped by the same means. The most reliable method of incorporation appeared to be potential cycling. It should be pointed out. however, that this is not a simple diffusion effect since a

preformed film cycled in a solution of substrate was found to lose its activity very quickly when transferred to a solution not containing the substrate. Iron and manganese tetra-phenyl porphyrins could also be incorporated and it is in these species that we are interested regarding catalysis. The reduced forms of these porphyrins are known to catalyze oxygen insertion reactions[14,15] but the reaction is impeded by the apparent parasitic formation of the μ-oxodimer. However, with the porphyrin constrained in a polymer matrix, such reactions are not possible. Figure 11 shows the $Fe^{III}/$ Fe^{II} redox couple obtained for PPP bound iron tetra-phenyl porphyrin in an oxygen saturated solution. Although the waves are well separated (presumably because of the film resistance) the re-oxidation wave is quite clearly visible. If the porphyrin were dissolved in solution, the reduction would be irreversible. Preliminary results indicated that a porphyrin bound in this way can catalyze the oxygenation of cyclohexene through quantitative measurements have not yet been made. Similar results were reported by Bull et al.[8a] for the reduction of oxygen by a phthalocyanine.

The ease with which films of PPP can be formed also leads us to consider the possible battery applications of the electroformed polymer film. Homogeneously polymerized phenylene has already been shown to have great promise in this area.[16] A simple cell was made by polymerizing PPP on two platinum electrodes and applying a constant voltage between them, while immersed in solution of $LiAsF_6$ in acetonitrile, for a short time. It was considered that in this way one of the electrodes would intercalate Li^- and become "n-type" while the other would become "p-type" and intercalate AsF_6^-. The cell voltage recorded under open circuit and on discharge is shown in Figure 12. The initial value was 3.8 V and this fell quickly to 0.2 V after circuit discharge through 1kohm.

DISCUSSION

A common goal among workers in the area of conducting polymers is polymer processibility. All literature reports on PPP indicate that the homogeneously prepared polymer is insoluble in all common solvents. The solubility of the electrochemically prepared version may be due to selective linking of shorter oligomers or to the presence of a quantity of m-linked polymer. m-linking gives rise to a more localized electronic structure increasing solubility but decreasing conductivity.[3] Nevertheless the polymer is conducting and soluble. The only other processible conducting polymer reported to date is a polyacetylene preparation.[17]

The MSRS signal increases with potential, as it should do as the number of carriers increases, and indicates the generation of two absorbing states on oxidation. It is generally accepted that for PPP, conduction proceeds via polarons at low defect concentration and bipolarons at high defect concentration.[18] (Solitons are not expected for PPP because of the high energy associated with the quinoid structure.) The bipolaron levels reported for PPP are 0.6 - 0.9eV above the valence band and 0.6 -0.9eV below the conduction band.[18] The MSRS absorption seen at 620nm may therefore be an excitation from the valence band to a polaron level, whereas that at 380nm is probably band gap excitation. Excitation to the lower polaron level would not be seen over the wavelength region studied.

Electrochemical characterization of the PPP film is still underway. There is clear dependence of the voltammetry on film growth conditions, which probably reflects different film morphologies. For example, it is possible that for films grown at low formation potentials or in dilute solutions, the average chain

length is shorter than for the corresponding film grown in concentrated solution. leading to lower conductivities. Electron microscopy would clearly be useful in this area. The impedance spectra clearly show that the film conductivity increases when the film is oxidized. The exact nature of the irreversible oxidation of the film at ~ 1.7V is not characterized; there is clearly. however. a marked reduction in conductivity. Further IR spectroscopic investigations may provide a clue to this reaction.

It is also unclear by what mechanism reactive species are trapped within the polymer. In an early paper Svanholm and Parker[19] discussed electrochemical "arylation" reactions which take place when species such as anthracene are oxidized in benzene. It may well be that the trapped species is actually chemically linked to the polymer by similar reaction with the radical. It is a little surprising that the porphyrin should show any redox activity since it is reduced at potentials where the film is not conducting. However. if the density of trapped porphyrin is sufficiently high. the porphyrin centers themselves may mediate charge transfer across the film. Digital simulations of these processes are being developed in these laboratories, and will provide the expected current transients and voltammetry for a variety of possible transport mechanisms.

The voltage generated in the PPP "battery" fell in a number of stages on discharge. This is almost certainly due to a distribution of chain lengths in the polymer. The shorter chains would have a higher oxidation potential than the longer chains and so would discharge first. There is apparently a solvent stability problem regarding cells made of n- and p-type PPP[20] which suggests that. in the experiment described above. electroplated Li may be one electrode in the cell.

CONCLUSIONS

PPP can be electrochemically generated from biphenyl in common laboratory solvents. The film thus formed is conducting in the oxidized state and insulating when neutral and can be dissolved in N-methyl pyrrolidone. Electrochemically grown PPP has been shown to incorporate solution free species present in the cell and to have potential use in battery applications.

ACKNOWLEDGEMENTS

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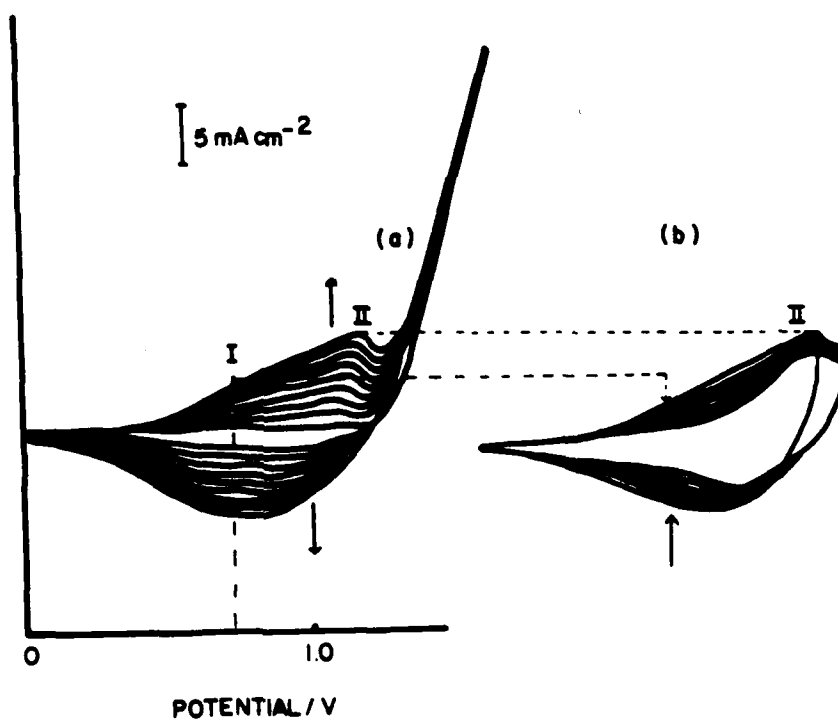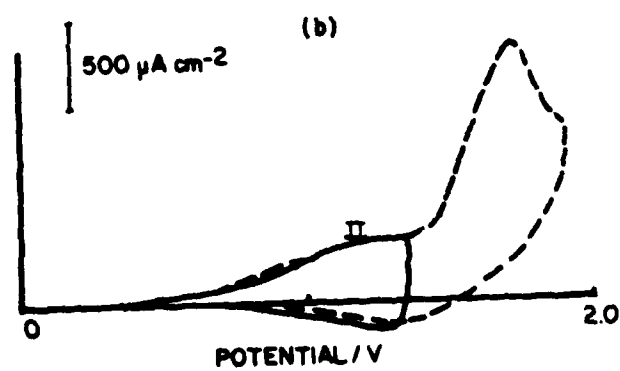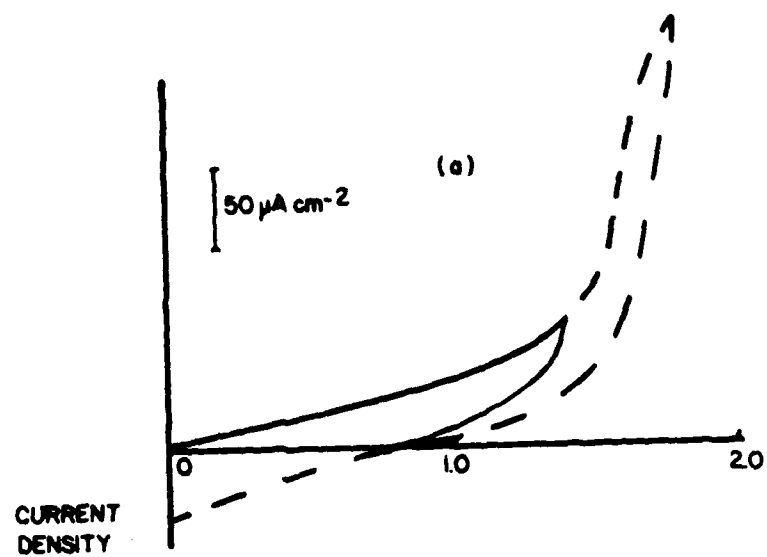
LEGENDS FOR THE FIGURES

1   Cyclic voltammograms recorded at a Pt electrode in a 1.0mM biphenyl solution in acetonitrile. The supporting electrolyte was tetra-n-butylammonium tetrafluoroborate (TBAF). Potentials vs. the Ag/Ag^+ (0.01M silver nitrate in 0.1M TBAF in acetonitrile) reference electrode. Note the rapid decrease in the magnitude of the current with continued cycling.

2   Cyclic voltammogram recorded at Pt electrode in a 1.0M biphenyl solution in acetonitrile/TBAF. Potentials vs. the Ag/Ag^+ (0.01M silver nitrate in 0.1M TBAF in acetonitrile) reference electrode. (a) Scans to potentials that force ohmic control. (b) Scans to regions remaining under diffusion control.

3   Cyclic voltammetry of "thin" (a) and "thick" (b) polyphenylene films in a solution containing only the supporting electrolyte (TBAF). The broken line shows the irreversible oxidation at 1.7V. Potentials vs. the Ag/Ag^- (0.01M silver nitrate in 0.1M TBAF in acetonitrile) reference electrode.

4   Potentiostatic growth of poly-paraphenylene showing the apparent nucleation of the polymer. Potentials vs. the Ag/Ag^+ (0.01M silver nitrate in 0.1M TBAF in acetonitrile) reference electrode.

5   In-phase and quadrature components of the impedance of a PPP film grown potentiostatically at 1.4V in a 0.1M biphenyl solution. Potentials vs. the Ag/Ag^- (0.01M silver nitrate in 0.1M TBAF in acetonitrile) reference electrode. C_p and R_p are the capacitative and resistive components of the impedance, respectively. The modulation frequencr is 80Hz.

6   Complex impedance diagrams for a PPP film grown potentiostatically at 1.3 V at (a) 0V, (b) +0.5mV, (c) +1.0V, (d) +2.0V, (e)1.0 V · Potentials vs. the Ag/Ag^+ (0.01M silver nitrate in 0.1M TBAF in acetonitrile) reference electrode. The film formation charge was 100mC cm^{-2}. Frequency range 500mHz to 10kHz. Z_{Re} is the resistive component of the impedance, while Z_{Im} is related to the capacitative component of the impedance.

7   IR reflectance spectra of (a) a neutral and (b) an oxidized PPP film.

8   MSRS of the same PPP film as reported in Figure 6. The applied potential was (a) 0-200mV. (b) 200-400mV and (c) 400-600mV at a frequency of 80Hz.

9   UV/VIS adsorption spectrum of the PPP film dissolved in N-methylpyrrolidone.

10 Cyclic voltammetry of a PPP film grown by potential cycling in the presence of ferrocene, showing the ferrocene/ferrocinium couple. Peak A is the oxidation of ferrocene to ferrocenium. while peak B corresponds to the reduction of ferrocenium to ferrocene.

11 Cyclic voltammetry showing the Fe^{III}/Fe^{II} couple for iron tetra-phenyl porphyrin trapped in a PPP film. The voltammogram was recorded in O_2 saturated acetonitrile/TBAF.

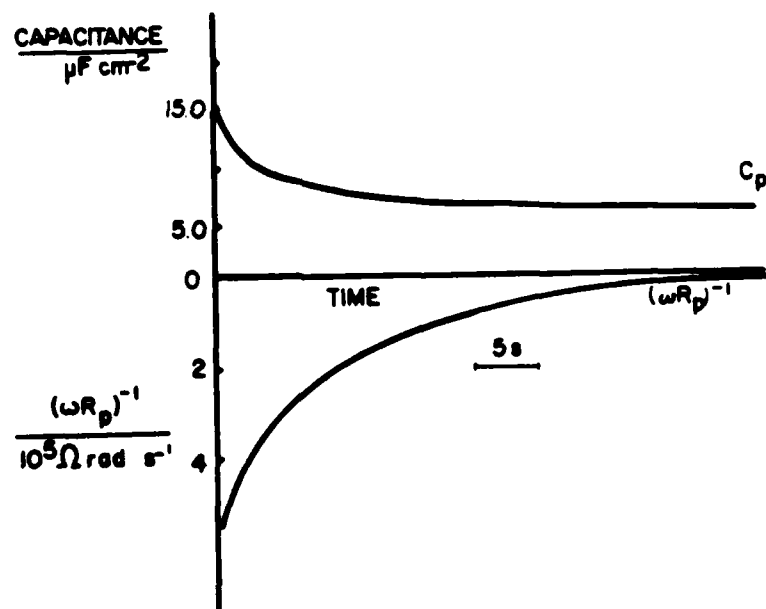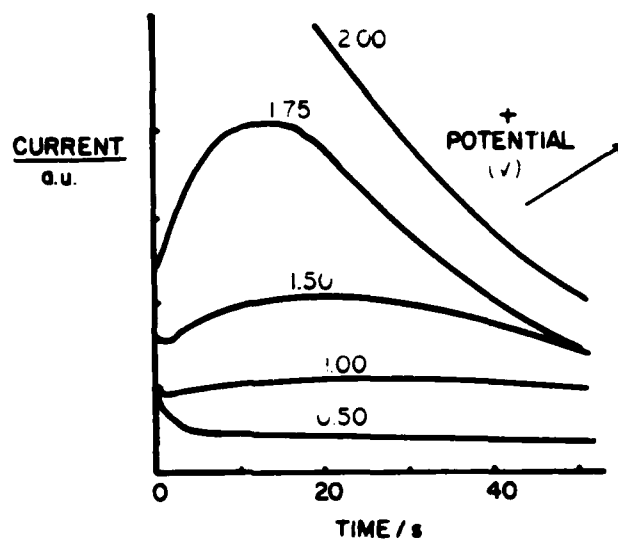12 Discharge characteristics of a simple PPP battery made by polarizing two PPP film electrodes in acetonitrile/$LiAsF_6$.
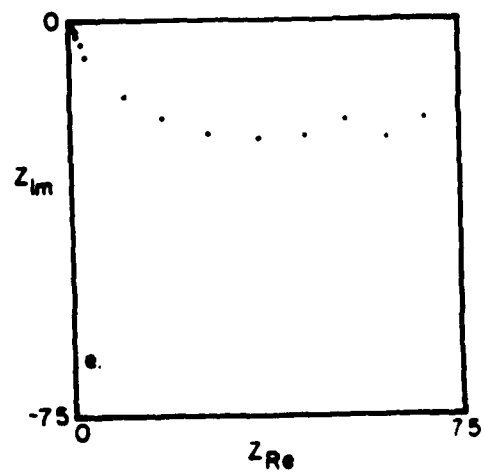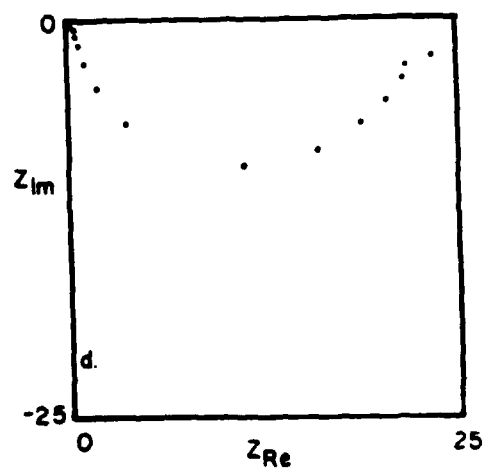
CURRENT
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POTENTIAL / V

CURRENT
DENSITY

$5 \, mA \, cm^{-2}$

(a)

(b)

I

II

II

POTENTIAL / V

WAVENUMBERS

ABSORBANCE

WAVELENGTH /nm

300   400

10×10^{-3}] $\Delta R/R$

$\Delta R/R$

a

b

c

400   600

WAVELENGTH / nm

CURRENT
a.u.

A

−0.6   O   0.7   E / V

B

VI

CURRENT

−0.8V   POTENTIAL

DISCHARGE THROUGH 1KΩ

4

CELL
POTENTIAL
VOLTS

40 s

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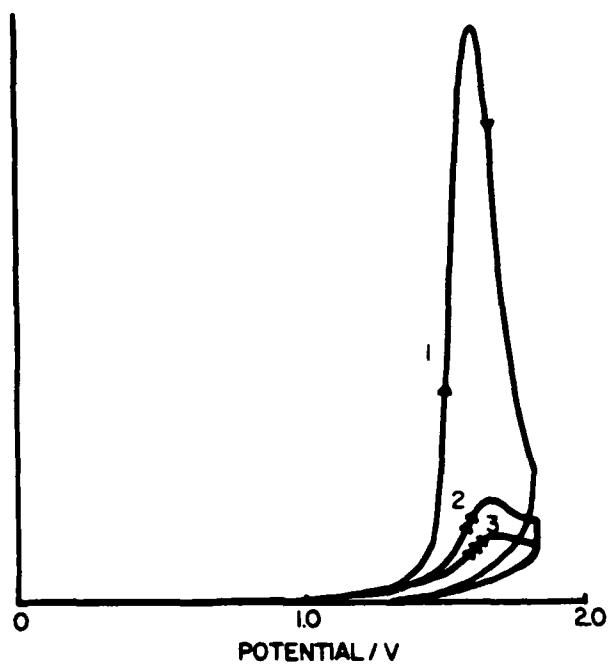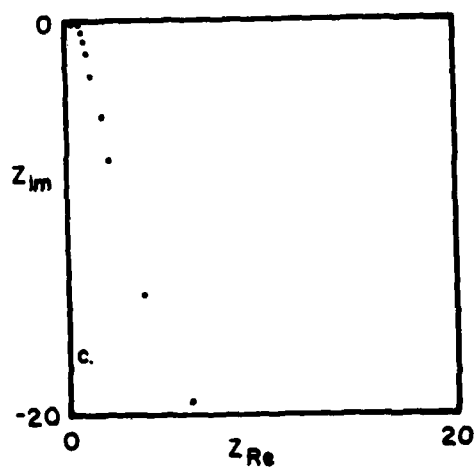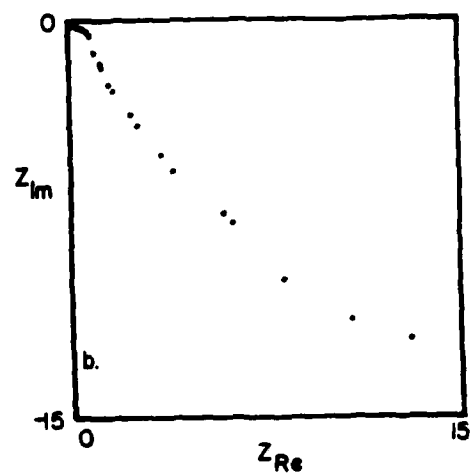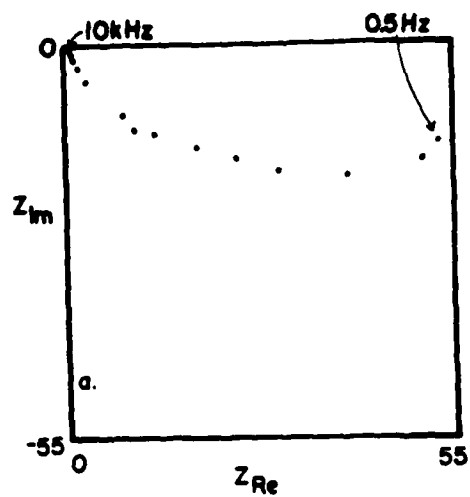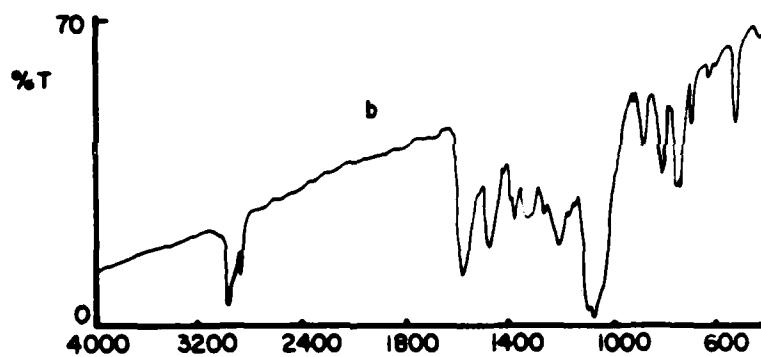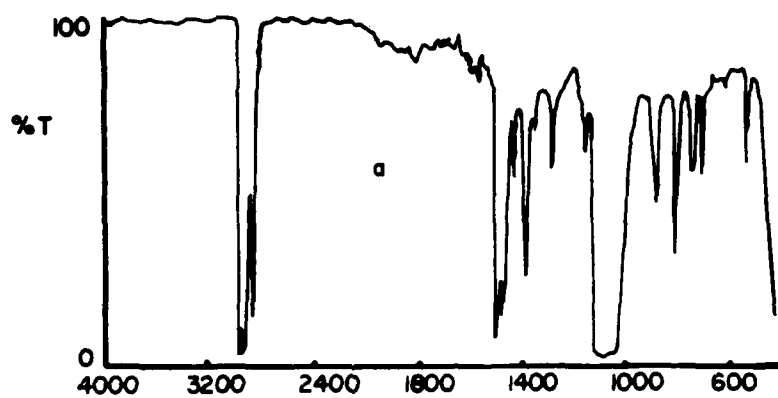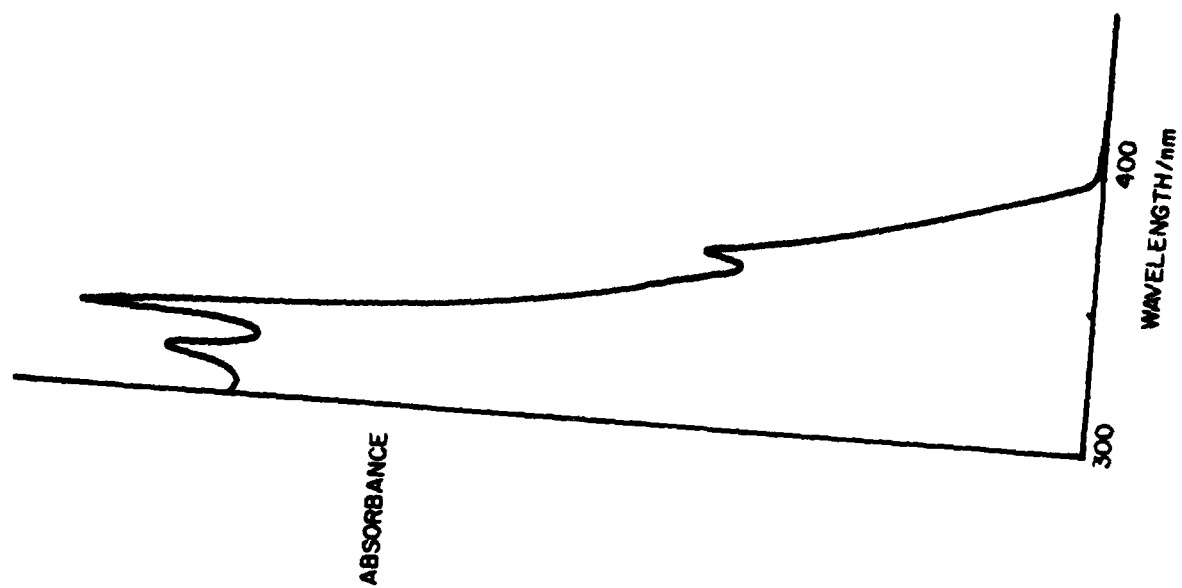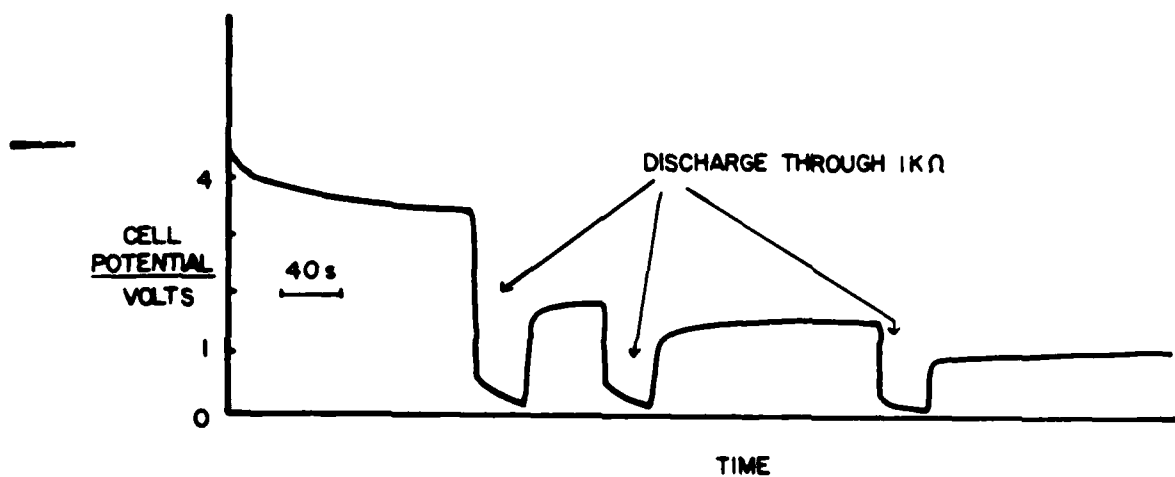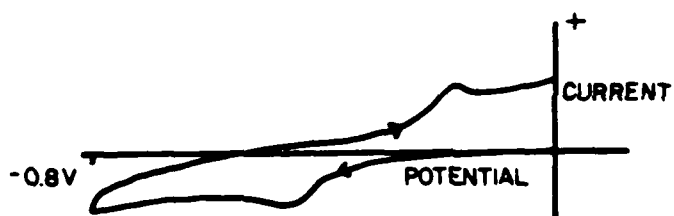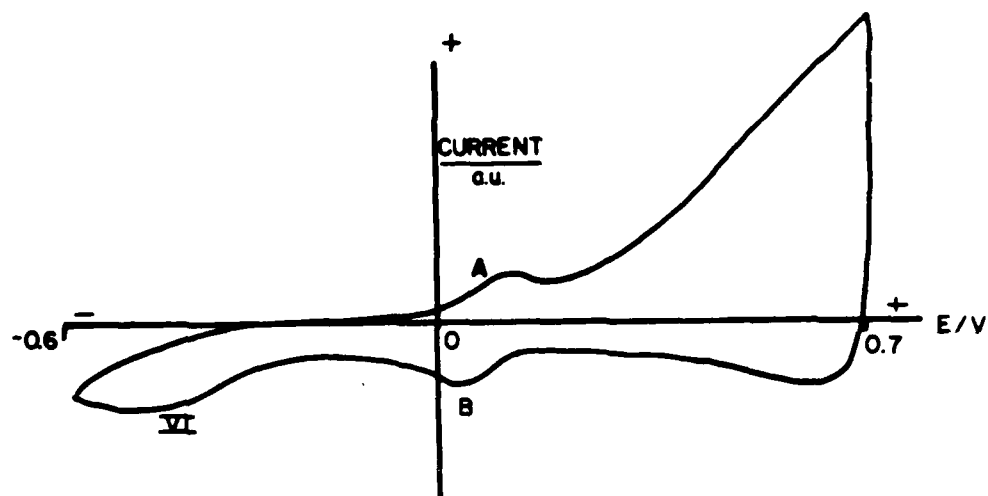
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